α-Hydroxycyclopentanones via one-pot oxidation of the trimethylaluminum-mediated Nazarov reaction with triplet oxygen

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Supporting Information: Experimental procedures, physical data and NMR spectra for all new compounds.

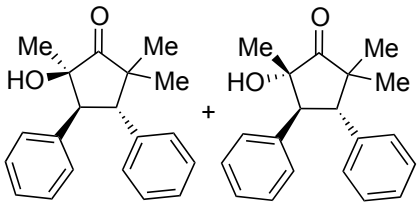
Table of Contents

General Information. Reactions were carried out in flame-dried glassware under a positive argon atmosphere unless otherwise stated. Transfer of anhydrous solvents and reagents was accomplished with oven-dried syringes or cannulae. 4Å molecular sieves were stored in ovens, and flame-dried before use. Solvents were distilled before use: dichloromethane from calcium hydride, tetrahydrofuran and toluene from sodium/benzophenone ketyl. Thin layer chromatography was performed on glass plates precoated with 0.25 mm Kieselgel 60 F254 (Merck). Flash chromatography columns were packed with 230-400 mesh silica gel (Silicycle). Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded at 400 MHz or 500 MHz and coupling constants (*J*) are reported in Hertz (Hz). Standard notation is used to describe the multiplicity of signals observed in ^{1}H NMR spectra: broad (br), apparent (app), multiplet (m), singlet (s), doublet (d), triplet (t), etc. Carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at 100 MHz or 125 MHz and are reported (ppm) relative to the center line of the triplet from chloroform-*d* (77.06 ppm). Infrared (IR) spectra were measured with a Mattson Galaxy Series FT-IR 3000 spectrophotometer. High-resolution mass spectrometry (HRMS) data

(APPI/APCI/ESI technique) were recorded using an Agilent Technologies 6220 oaTOF instrument. HRMS data (EI technique) were recorded using a Kratos MS50 instrument. Extra dry oxygen (99.6%, H_2O < 10ppm) from Praxair was used for the oxidation.
Dienones **1a**,[1] **1b**,[2] **1c**,[3] **1d**,[4] **1e**,[3] **1f**,[5] **1g**,[6] and **1h**,[7] were prepared via literature procedures.

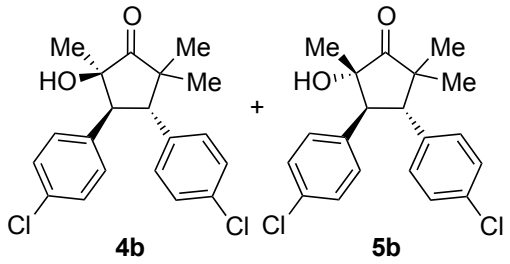
Representative procedure of the construction of α-hydroxycyclopentanones (4a/5a): 4Å Activated 4Å molecular sieves (100 mg) were suspended in a solution of **1a** (50 mg, 0.19 mmol) in CH_2Cl_2 (1.9 mL, 0.1M). The mixture was cooled to –41ºC and $AlMe_3$ (0.24 mL, 2.0 M solution in toluene) was added dropwise. The reaction mixture was stirred at –41ºC until complete consumption of **1a** was observed by TLC (30 min). The solution was purged with O_2 gas for 5 min at –41ºC, then the solution was allowed to warm to 0 ºC under a static oxygen atmosphere and stirred for 2 hrs. The reaction was quenched with 1M aq. HCl (3 mL) and warmed to room temperature. After separation of the phases, the aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic extracts were washed with brine, and dried over $MgSO_4$, filtered, and concentrated in vacuo. Purification by flash column chromatography (silica gel) provided the desired products **4a** (25 mg, 45 %) and **5a** (19 mg, 34%).

Spectral Data of 4a/5a to 4g/5g, and 6h/7h



4a **5a**

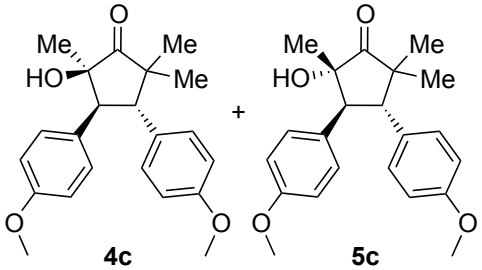
Reaction was performed under the standard procedure. Flash chromatography (19:1 to 8:2 hexane:EtOAc) gave **4a** (25 mg, 45 %) and **5a** (19 mg, 34 %) as white solids.
4a: R_f 0.42 (hexanes/EtOAc 8:2); mp 188-190 °C; IR (cast film) 3442, 3026, 2982, 1737, 1449, 1394 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.32-7.13 (m, 10H), 3.89 (d, *J* = 13.2 Hz, 1H), 3.53 (d, *J* = 13.2 Hz, 1H), 1.57 (br, 1H), 1.37 (s, 3H), 1.33 (s, 3H), 0.75 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 219.3 136.9, 135.3, 129.8, 129.0, 128.3, 128.1, 127.3, 126.8, 76.4, 53.1, 52.7, 48.5, 24.5, 22.4, 20.6; HRMS (EI, M^+) for $C_{20}H_{22}O_2$ calcd. 294.1620, found: m/z 294.1619.
5a: R_f 0.33 (hexanes/EtOAc 8:2); mp 191-194 °C; IR (cast film) 3426, 3030, 2972, 1739, 1498, 1451 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.30-7.16 (m, 10H), 3.96 (d, *J* = 13.5 Hz, 1H), 3.54 (d, *J* = 13.5 Hz, 1H), 2.75 (s, 1H), 1.32 (s, 3H), 0.99 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 223.1, 136.7, 136.3, 128.9, 128.9, 128.3, 128.2, 126.9, 126.9, 79.9, 51.4, 51.1, 47.3, 24.9, 21.5, 20.7; HRMS (EI, M^+) for $C_{20}H_{22}O_2$ calcd. 294.1620, found: m/z 294.1616.



Dienone **1b** (53 mg, 0.16 mmol) was stirred with $AlMe_3$ for 1 h at –41 ºC, and then was stirred for 3 h at 0 ºC under O_2 atmosphere. Flash chromatography (19:1 to 8:2 hexane:EtOAc) gave **4b** (22 mg, 38 %) and **5b** (18 mg, 31 %) as white solids.

4b: R_f 0.34 (hexanes/EtOAc 8:2); mp 152-154 °C; IR (cast film) 3462, 2969, 1741, 1494, 1092, 760 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.25-7.22 (m, 6H), 7.10-7.08 (m, 2H), 3.82 (d, *J* = 13.2 Hz, 1H), 3.42 (d, *J* = 13.2 Hz, 1H), 1.70 (s, 1H), 1.37 (s, 3H), 1.32 (s, 3H), 0.77 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 218.7 135.1, 133.6, 133.3, 132.8, 131.0, 130.1, 128.5, 128.5, 76.2, 52.7, 52.6, 48.3, 24.5, 22.2, 20.4; HRMS (EI, M^+) for $C_{20}H_{20}O_2{}^{35}Cl_2$ calcd. 362.0841, found: m/z 362.0840.

5b: R_f 0.23 (hexanes/EtOAc 8:2); mp 145-147 °C; IR (cast film) 3449, 2970, 1746, 1494, 1092, 756 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.28-7.23 (m, 4H), 7.20-7.18 (m, 2H), 7.14-7.11 (m, 2H), 3.86 (d, *J* = 13.5 Hz, 1H), 3.45 (d, *J* = 13.5 Hz, 1H), 2.77 (s, 1H), 1.30 (s, 3H), 0.97 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 222.3, 134.9, 134.5, 133.1, 133.0, 130.2, 130.1, 128.8, 128.5, 79.7, 51.0, 50.7, 47.3, 24.8, 21.4, 20.6; HRMS (EI, M^+) for $C_{20}H_{20}O_2{}^{35}Cl_2$ calcd. 362.0841, found: m/z 362.0835.

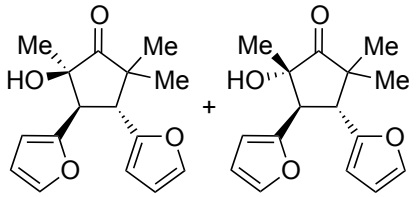


Dienone **1c** (51 mg, 0.15 mmol) was stirred with $AlMe_3$ for 1 h at –41 ºC, and then was stirred for 2 h at 0 ºC under O_2 atmosphere. Flash chromatography (19:1 to 8:2 hexane:EtOAc) gave **4c** (22 mg, 41 %) as a white solid, and **5c** (19 mg, 36 %) as a colorless oil.

4c: R_f 0.47 (hexanes/EtOAc 2:1); mp 130-133 °C; IR (cast film) 3469, 2965, 2932, 2836, 1742, 1514, 1248 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.25-7.22 (m, 2H), 7.10-7.07 (m, 2H), 6.84-6.81 (m, 2H), 6.80-6.77 (m, 2H), 3.78 (d, *J* = 13.2 Hz, 1H), 3.76 (s, 3H), 3.75 (s, 3H), 3.45 (d, *J* = 13.2 Hz, 1H), 1.57 (s, 1H), 1.37 (s, 3H), 1.31 (s, 3H), 0.76 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 219.7 158.8, 158.4, 130.7, 129.9, 129.0, 127.2, 113.8, 113.6, 76.3, 55.1, 55.1, 52.6, 52.2, 48.5, 24.5, 22.5, 20.5; HRMS (EI, M^+) for $C_{22}H_{26}O_4$ calcd. 354.1831, found: m/z 354.1832.

5c: R_f 0.35 (hexanes/EtOAc 2:1); IR (film) 3464, 2966, 1743, 1515, 1249 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.20-7.17 (m, 2H), 7.13-7.10 (m, 2H), 6.83-6.78 (m, 4H), 3.84 (d, *J* = 13.5 Hz, 1H), 3.77 (s, 3H), 3.75 (s, 3H), 3.43 (d, *J* = 13.4 Hz, 1H), 2.73 (s, 1H), 1.29 (s, 3H), 0.97 (s, 3H),

0.79 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 223.5, 158.5, 158.4, 129.9, 129.8, 128.7, 128.4, 113.8, 113.7, 80.0, 55.2, 55.1, 51.0, 50.6, 47.3, 24.9, 21.4, 20.7; HRMS (EI, M^+) for $C_{22}H_{26}O_4$ calcd. 354.1831, found: m/z 354.1838.

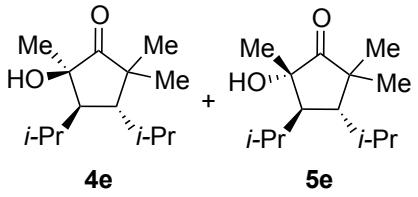


4d **5d**

Dienone **1d** (50 mg, 0.21 mmol) was stirred with $AlMe_3$ for 1 h at –41 ºC, and then was stirred for 3 h at 0 ºC under O_2 atmosphere. Flash chromatography (19:1 to 8:2 hexane:EtOAc) gave **4d** (21 mg, 36 %) and **5d** (16 mg, 28 %) as white solids.

4d: R_f 0.34 (hexanes/EtOAc 8:2); mp 131-133 °C; IR (cast film) 3428, 2973, 2933, 1740, 1505, 1147, 1006, 724 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 1.8, 0.8 Hz, 1H), 7.34 (dd, *J* = 1.9, 0.8 Hz, 1H), 6.34 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.29 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.21 (ddd, *J* = 3.3, 0.7, 0.7 Hz, 1H), 6.08 (ddd, *J* = 3.2, 0.7, 0.7 Hz, 1H), 3.78 (d, *J* = 12.8 Hz, 1H), 3.62 (d, *J* = 12.9 Hz, 1H), 1.92 (s, 1H), 1.46 (s, 3H), 1.38 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 217.8 152.8, 150.9, 142.3, 141.8, 110.5, 110.1, 108.4, 107.4, 76.2, 48.4, 47.0, 47.0, 24.7, 22.3, 20.8; HRMS (EI, M^+) for $C_{16}H_{18}O_4$ calcd. 274.1205, found: m/z 274.1203.

5d: R_f 0.20 (hexanes/EtOAc 8:2); mp 158-160 °C; IR (cast film) 3437, 2975, 1737, 1501, 1365, 1012, 742, 727 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 1.9, 0.8 Hz, 1H), 7.32 (dd, *J* = 1.8, 0.8 Hz, 1H), 6.29 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.27 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.14 (ddd, *J* = 3.2, 0.8, 0.8 Hz, 1H), 6.06 (ddd, *J* = 3.2, 0.8, 0.8 Hz, 1H), 3.82 (d, *J* = 13.1 Hz, 1H), 3.51 (d, *J* = 13.1 Hz, 1H), 2.71 (s, 1H), 1.33 (s, 3H), 1.07 (s, 3H), 0.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 221.7 152.4, 151.7, 142.1, 141.9, 110.2, 110.2, 108.0, 107.4, 79.8, 47.4, 46.7, 45.6, 25.1, 21.3, 21.1; HRMS (EI, M^+) for $C_{16}H_{18}O_4$ calcd. 274.1205, found: m/z 274.1204.



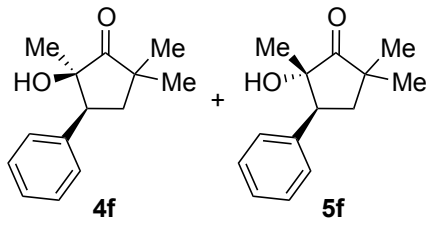
4e **5e**

Dienone **1e** (74 mg, 0.38 mmol) was stirred with $AlMe_3$ for 2 h at –25 ºC,[8] and then was stirred for 3 h at 0 ºC under O_2 atmosphere. Flash chromatography (49:1 to 9:1 hexane:EtOAc) gave **4e** (11 mg, 13 %) and **5e** (18 mg, 20 %) as white solids.

4e: R_f 0.35 (hexanes/EtOAc 9:1); mp 106-108 °C; IR (cast film) 3458, 2964, 2891, 1744, 1385, 1372 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.06 ([X of ABXY] app sept of d, *J* = 7.1, 3.3 Hz, 1H), 1.97 ([Y of ABXY] app sept of d, *J* = 7.1, 3.0 Hz, 1H), 1.91-1.85 (AB portion of ABXY, J_{AB} = 10.6 Hz, $J_{AX} = J_{BY}$ = 3.3 Hz, 2H), 1.78 (s, 1H), 1.36 (s, 3H), 1.18 (s, 3H), 1.12 (d, *J* = 7.0 Hz, 3H), 1.06 (d, *J* = 7.0 Hz, 3H), 1.04 (d, *J* = 7.1 Hz, 3H), 1.01 (d, *J* = 7.0 Hz, 3H), 0.99 (s, 3H); ^{13}C

NMR (125 MHz, $CDCl_3$) δ 224.2, 78.1, 50.1, 49.4, 47.9, 28.1, 27.5, 27.3, 26.0, 22.6, 21.6, 21.1, 20.2, 20.0; HRMS (EI, M^+) for $C_{14}H_{26}O_2$ calcd. 226.1933, found: m/z 226.1939.

5e: R_f 0.20 (hexanes/EtOAc 9:1); mp 112-114 °C; IR (cast film) 3446, 2964, 2888, 1741, 1391, 1366 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.53 (s, 1H), 2.13 (app sept of d, J = 7.3, 3.9 Hz, 1H), 2.08 (app sept of d, J = 7.3, 3.3 Hz, 1H), 2.04 (dd, J = 12.9, 3.8 Hz, 1H), 1.81 (dd, J = 12.9, 3.3 Hz, 1H), 1.23 (s, 3H), 1.19 (s, 3H), 1.18 (d, J = 7.2 Hz, 3H), 1.12 (d, J = 7.2 Hz, 3H), 1.09 (d, J = 7.3 Hz, 3H), 1.08 (s, 3H), 1.06 (d, J = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 225.9, 80.2, 49.8, 49.0, 46.8, 27.5, 27.1, 26.8, 22.1, 21.8, 20.9, 20.7, 20.7, 19.6; HRMS (EI, M^+) for $C_{14}H_{26}O_2$ calcd. 226.1933, found: m/z 226.1933.

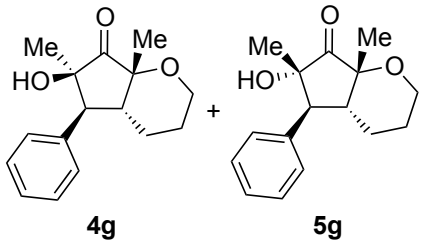


4f **5f**

Dienone **1f** (58 mg, 0.31 mmol) was stirred with $AlMe_3$ for 150 min at –41 ºC, and then was stirred for 3 h at 0 ºC under O_2 atmosphere. Flash chromatography (19:1 to 9:1 hexane:EtOAc) gave **4f** (22 mg, 32 %) and **5f** (24 mg, 36 %) as white solids.

4f: R_f 0.45 (hexanes/EtOAc 8:2); mp 116-118 °C; IR (cast film) 3431, 3029, 2972, 1742, 1455, 706 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.41-7.36 (m, 4H), 7.35-7.32 (m, 1H), 3.08 (dd, J = 13.1, 6.4 Hz, 1H), 2.46 (app. t, J = 12.8 Hz, 1H), 1.99 (dd, J = 12.4, 6.4 Hz, 1H), 1.56 (s, 1H), 1.34 (s, 3H), 1.27 (s, 3H), 1.18 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 220.2, 137.2, 129.5, 128.3, 127.4, 76.9, 50.0, 44.1, 40.4, 25.9, 25.3, 21.8; HRMS (EI, M^+) for $C_{14}H_{18}O_2$ calcd. 218.1307, found: m/z 218.1307.

5f: R_f 0.32 (hexanes/EtOAc 8:2); mp 130-131 °C; IR (cast film) 3432, 3029, 2969, 1739, 1364 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-7.36 (m, 4H), 7.33-7.29 (m, 1H), 3.50-3.44 (m, 1H), 2.77 (s, 1H), 2.15-2.06 (m, 2H), 1.28 (s, 3H), 1.25 (s, 3H), 0.90 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 224.3, 138.0, 128.3, 128.2, 127.0, 80.4, 48.1, 42.6, 37.7, 26.0, 26.0, 19.6; HRMS (EI, M^+) for $C_{14}H_{18}O_2$ calcd. 218.1307, found: m/z 218.1305.



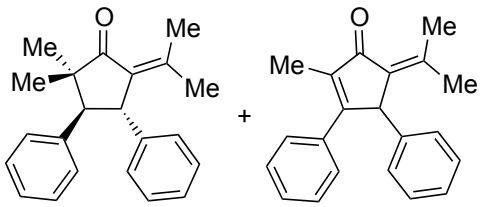
4g **5g**

Dienone **1g** (70 mg, 0.31 mmol) was stirred with $AlMe_3$ for 90 min at –41 ºC, and then was stirred for 4 h at 0 ºC under O_2 atmosphere. Flash chromatography (9:1 hexane:EtOAc) gave impure **4g**, and pure **5g** (25 mg, 31 %) as a white solid. Pure **4g** (colorless oil, 14mg, 18%) could

be obtained after an additional flash chromatographic purification (1:4:19 to 2:6:16 to 2:3:5 Et_2O: DCM: Hex).

4g: R_f 0.53 (hexanes/EtOAc 2:1); IR (film) 3488, 3027, 2933, 2861, 1759, 1453, 1374, 1054 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.42-7.38 (m, 2H), 7.36-7.32 (m, 3H), 3.83-3.79 (m, 1H), 3.72-3.67 (m, 1H), 3.43 (d, *J* = 12.9 Hz, 1H), 2.59-2.55 (m, 1H), 1.87-1.75 (m, 2H), 1.58-1.55 (m, 1H), 1.52 (br, 1H), 1.51 (s, 3H), 1.34 (s, 3H), 1.34-1.30 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 211.9 135.7, 129.9, 128.5, 127.5, 76.9, 76.2, 61.9, 51.6, 40.5, 22.0, 20.0, 18.6, 16.6; HRMS (EI, M^+) for $C_{16}H_{20}O_3$ calcd. 260.1412, found: m/z 260.1413.

5g: R_f 0.34 (hexanes/EtOAc 2:1); mp 176-178 °C; IR (cast film) 3449, 3030, 2942, 2859, 1753, 1370, 1057 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.42-7.38 (m, 2H), 7.34-7.27 (m, 3H), 3.86-3.82 (m, 1H), 3.77 (d, *J* = 13.2 Hz, 1H), 3.69 (ddd, *J* = 11.9, 11.9, 2.5 Hz, 1H), 2.57 (br, 1H), 2.32-2.28 (m, 1H), 1.93-1.75 (m, 3H), 1.48 (s, 3H), 1.37-1.32 (m, 1H), 0.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 215.9, 136.1, 128.9, 128.6, 127.3, 79.6, 75.6, 61.9, 50.7, 38.2, 20.8, 20.2, 18.6, 17.3; HRMS (EI, M^+) for $C_{16}H_{20}O_3$ calcd. 260.1412, found: m/z 260.1415.



6h **7h**

To a solution of **1h** (50 mg, 0.16 mmol) in DCM was added 2.5 equiv of $AlMe_3$ at 0 ºC. The reaction mixture was warmed to rt and stirred for 26 h and then an additional 16 h under O_2 atmosphere. Flash chromatography (49:1 to 24:1 hexane:EtOAc) gave **6h** (19 mg, 39 %) as a colorless oil, and **7h** (10 mg, 22 %) as a white solid.

6h: R_f 0.43 (hexanes/EtOAc 9:1); IR (film) 3028, 2965, 2929, 1704, 1623, 705 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.11-7.03 (m, 6H), 6.89-6.85 (m, 2H), 6.63-6.60 (m, 2H), 4.53-4.51 (m, 1H), 3.37 (d, *J* = 8.6 Hz, 1H), 2.43 (d, *J* = 1.8 Hz, 3H), 1.62 (d, *J* = 1.0 Hz, 3H), 1.17 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 211.3, 153.0, 141.3, 138.2, 132.5, 130.9, 129.6, 127.6, 127.3, 126.3, 126.0, 57.6, 50.5, 49.7, 26.6, 25.5, 22.3, 21.7; HRMS (EI, M^+) for $C_{22}H_{24}O$ calcd. 304.1827, found: m/z 304.1826.

7h: R_f 0.29 (hexanes/EtOAc 9:1); mp 138-141 °C; IR (cast film) 3026, 2919, 1679, 1623, 700 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.33-7.25 (m, 3H), 7.21-7.15 (m, 4H), 7.11-7.05 (m, 3H), 4.78 (br, 1H), 2.39 (d, *J* = 0.4 Hz, 3H), 1.98 (d, *J* = 1.8 Hz, 3H), 1.70 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.5, 162.4, 147.3, 140.9, 139.4, 135.3, 134.5, 128.5, 128.4, 128.3, 128.2, 128.1, 126.4, 52.5, 24.0, 20.7, 9.9; HRMS (EI, M^+) for $C_{21}H_{20}O$ calcd. 288.1514, found: m/z 288.1515.

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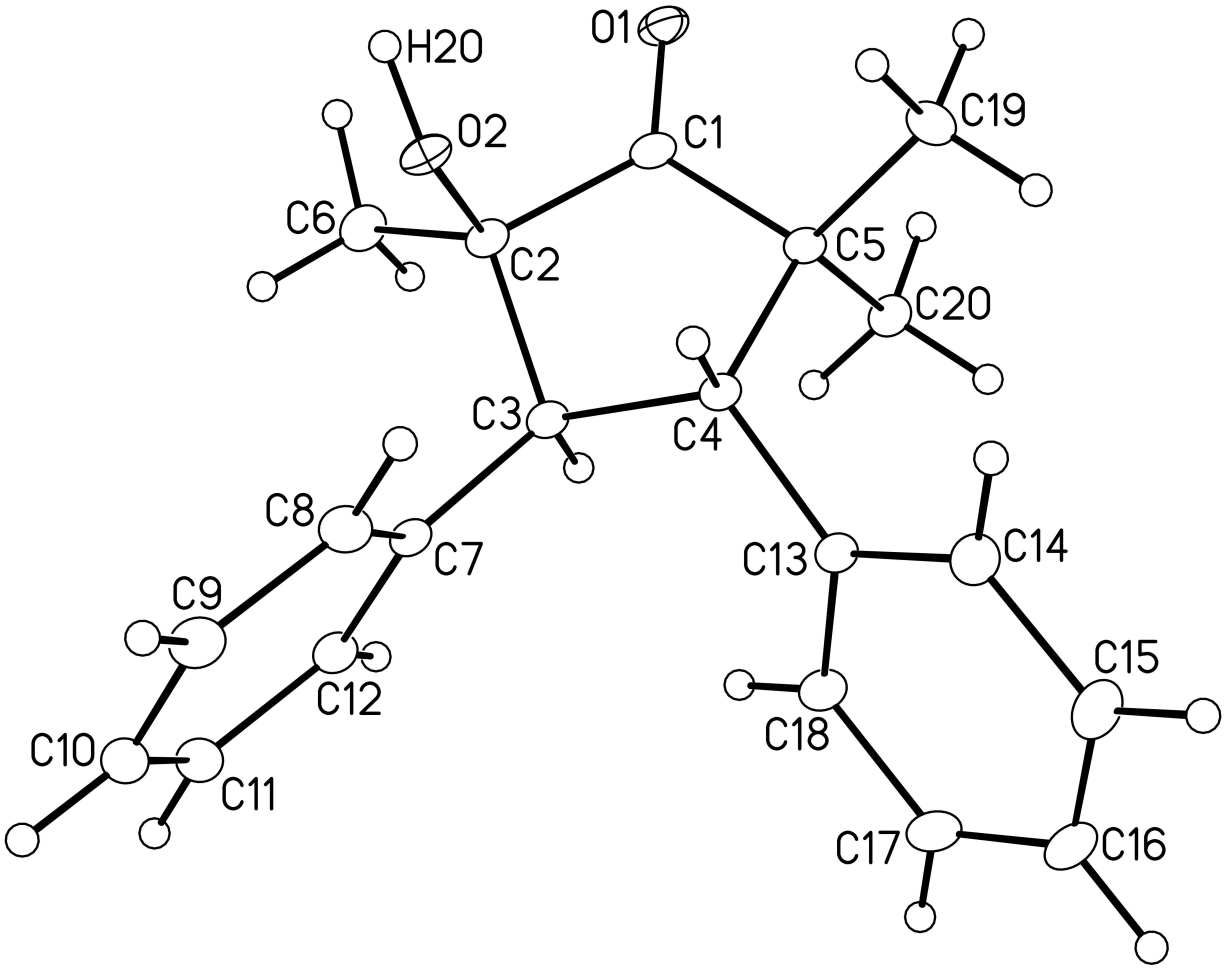
5) Y. Wang, B. D. Schill, A. M. Arif, F. G. West, *Org. Lett.* **2003**, *5*, 2747-2750.

6) G. Liang, S. N. Gradl, D. Trauner, *Org. Lett.* **2003**, *5*, 4931-4934.

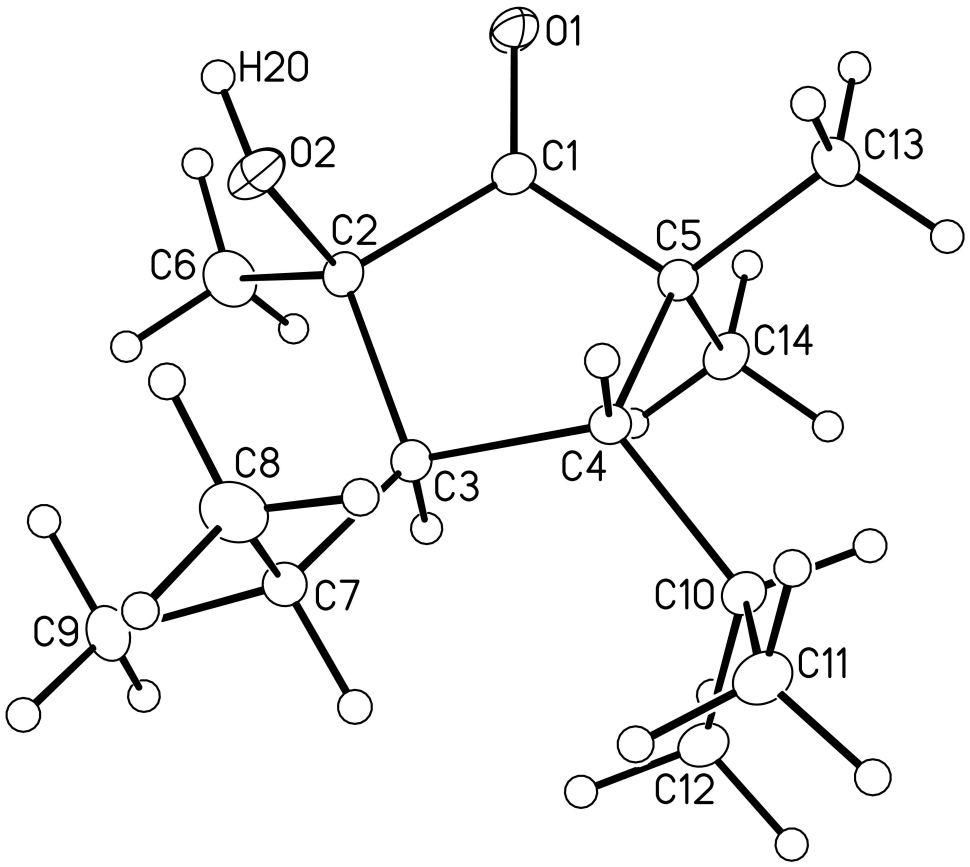
7) O. Scadeng, F. G. West, *Eur. J. Org. Chem.* **2014**, 1860–1865.

8) Ethylene glycol/ethanol bath with dry ice was used to obtain a stable bath temperature of –25 ºC. See: D. W. Lee, C. M. Jensen, *J. Chem. Educ.* **2000**, *77*, 629.

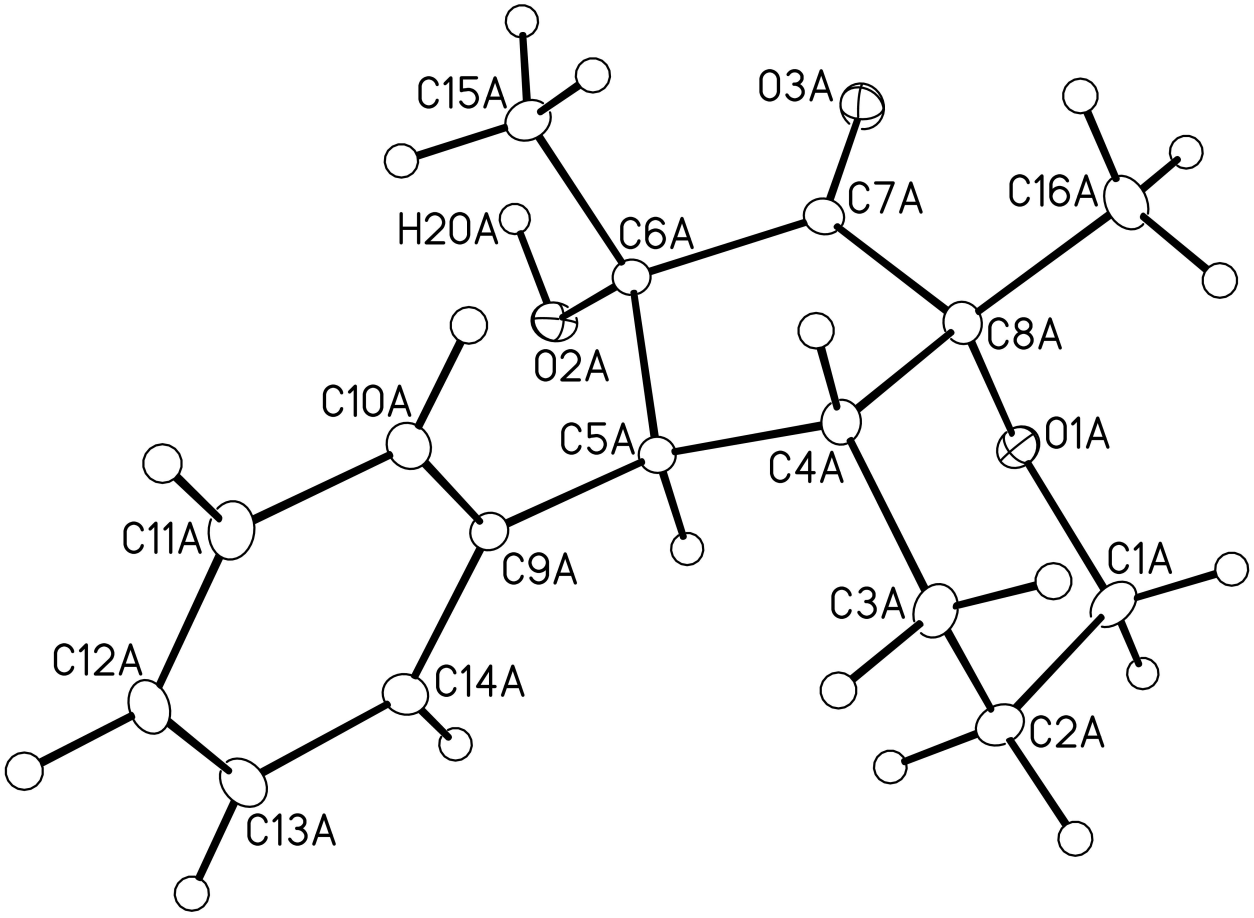
ORTEP Structure of 4a



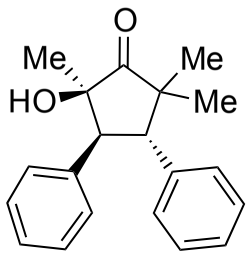
ORTEP Structure of 4e



ORTEP Structure of 5g

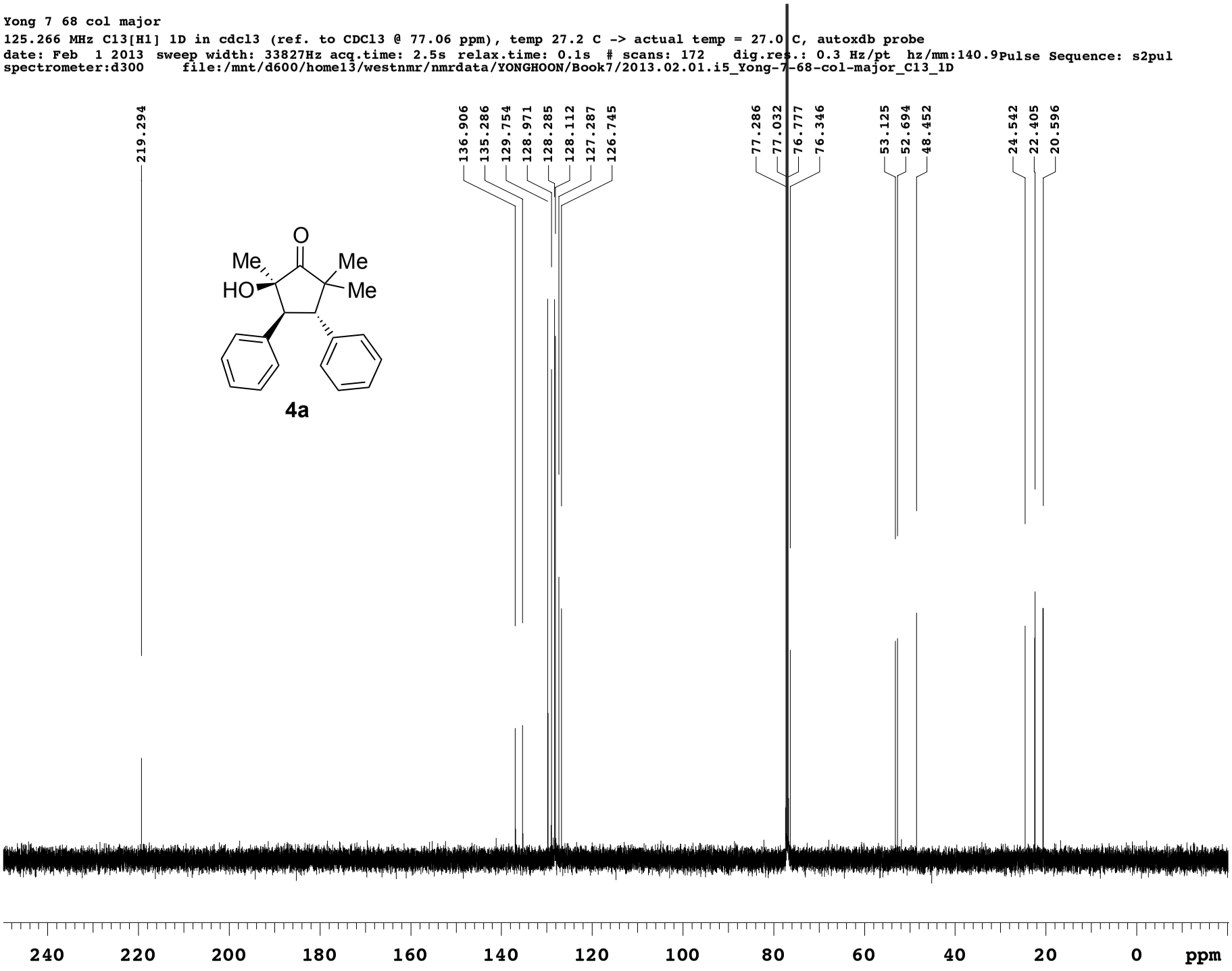


Yong 5 134 col 2ndspot
498.122 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Jun 5 2012 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.3 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book5/2012.06.05.i5_Yong-5-134-col-2ndspot_H1_1D

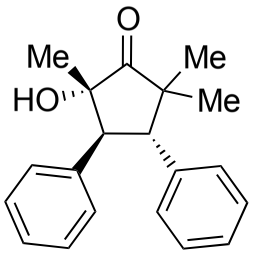


4a

Yong 7 68 col major
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 1 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 172 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.01.i5_Yong-7-68-col-major_C13_1D

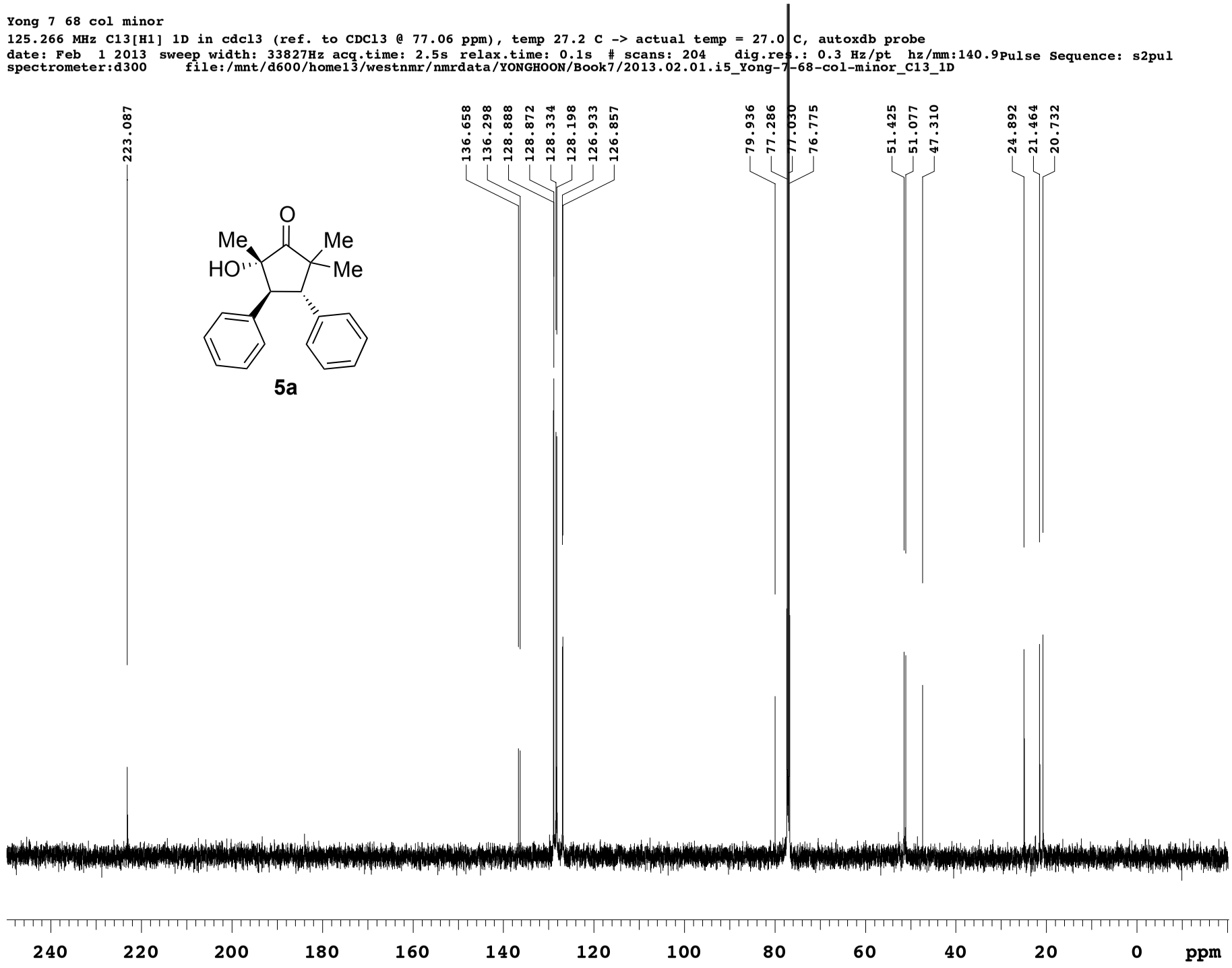


Yong 7 68 col minor solvent removed
399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, autoxdb probe
date: Apr 2 2013 sweep width: 4802Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:15.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.04.02.i4_Yong-7-68-col-minor-solvent_removed_H1_1D



5a

Yong 7 68 col minor
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 1 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 204 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.01.i5_Yong-7-68-col-minor_C13_1D

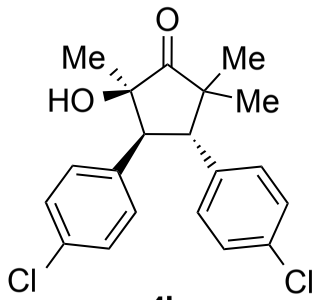


Yong 7 98 col 1stspot EAremoved

498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

date: Feb 19 2013 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:25.0 Pulse Sequence: s2pul

spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.19.i5_Yong-7-98-col-1stspot-EAremoved_H1_1D



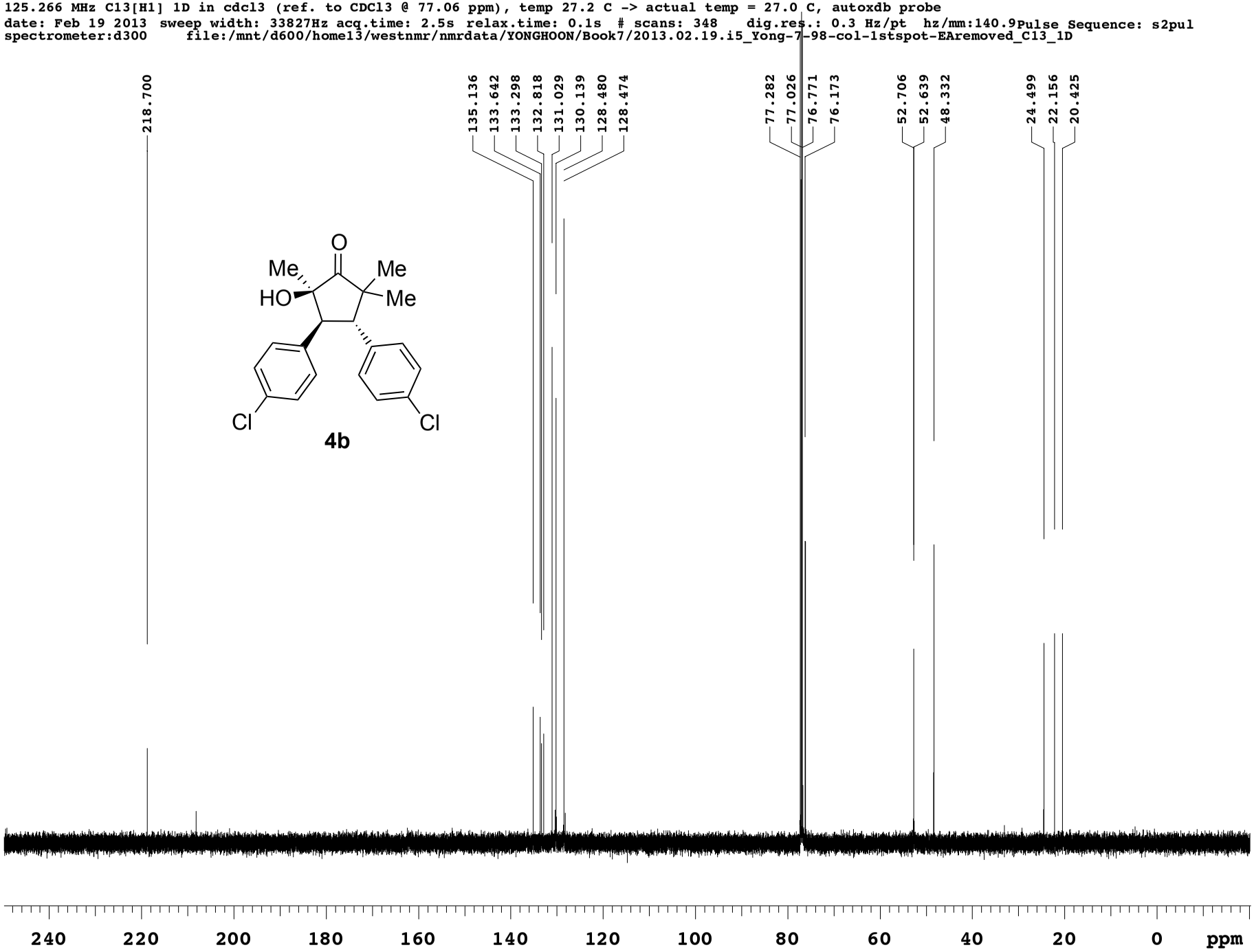
4b

Yong 7 98 col 1stspot EAremoved

125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

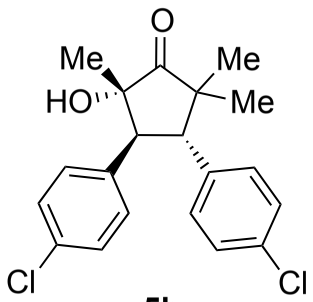
date: Feb 19 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 348 dig.res.: 0.3 Hz/pt hz/mm:140.9Pulse Sequence: s2pul

spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.19.i5_Yong-7-98-col-1stspot-EAremoved_C13_1D

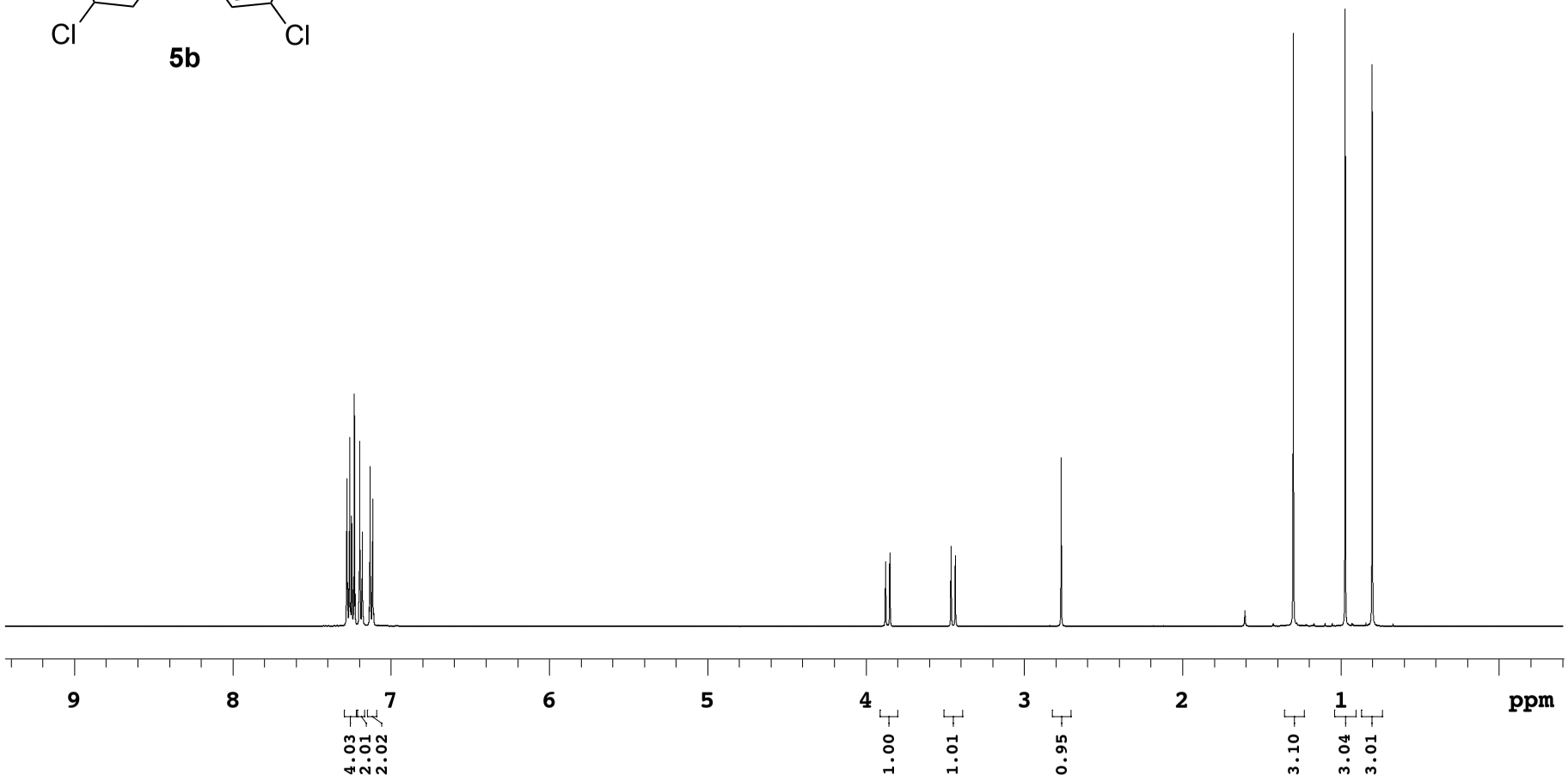


Yong-7-158-col-2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, autoxdb probe

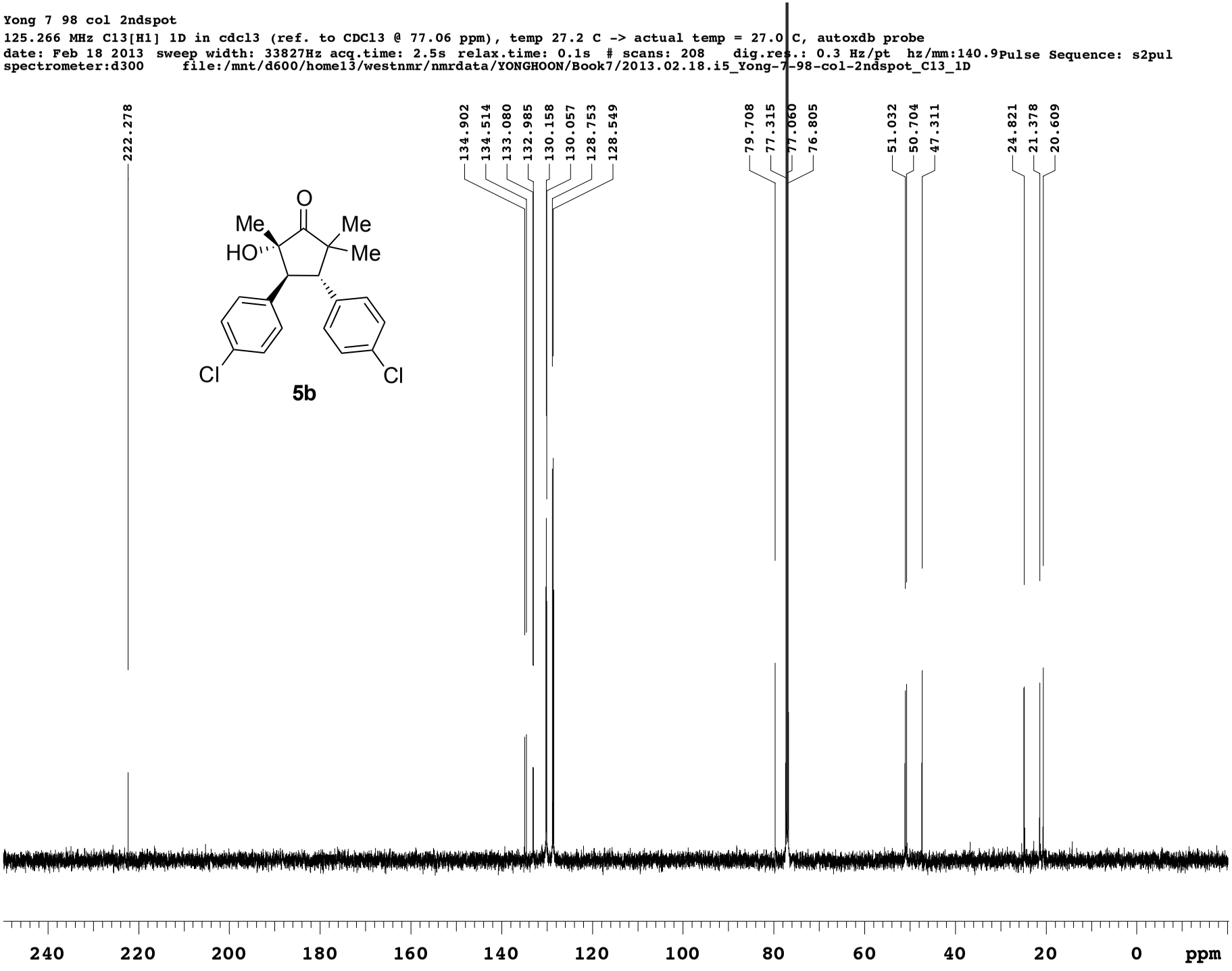
Pulse Sequence: s2pul



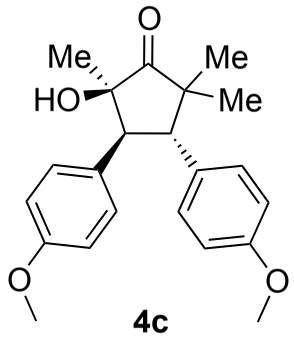
5b



Yong 7 98 col 2ndspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 18 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 208 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.18.i5_Yong-7-98-col-2ndspot_C13_1D

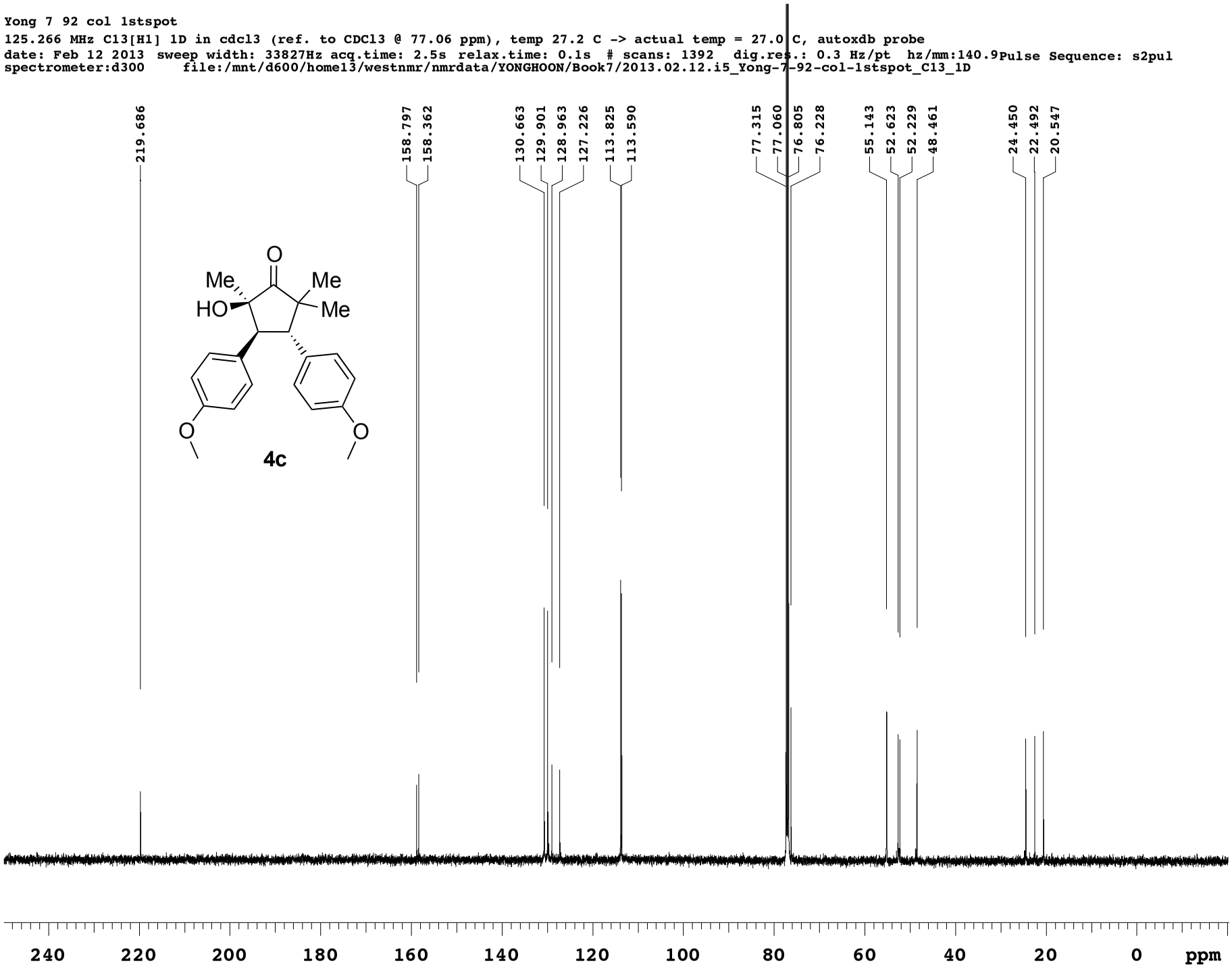


Yong 7 92 col 1stspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 12 2013 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.4 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.12.i5_Yong-7-92-col-1stspot_H1_1D



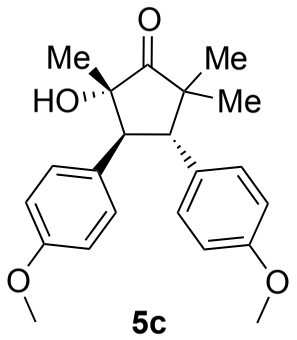
4c

Yong 7 92 col 1stspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 12 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 1392 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.12.i5_Yong-7-92-col-1stspot_C13_1D



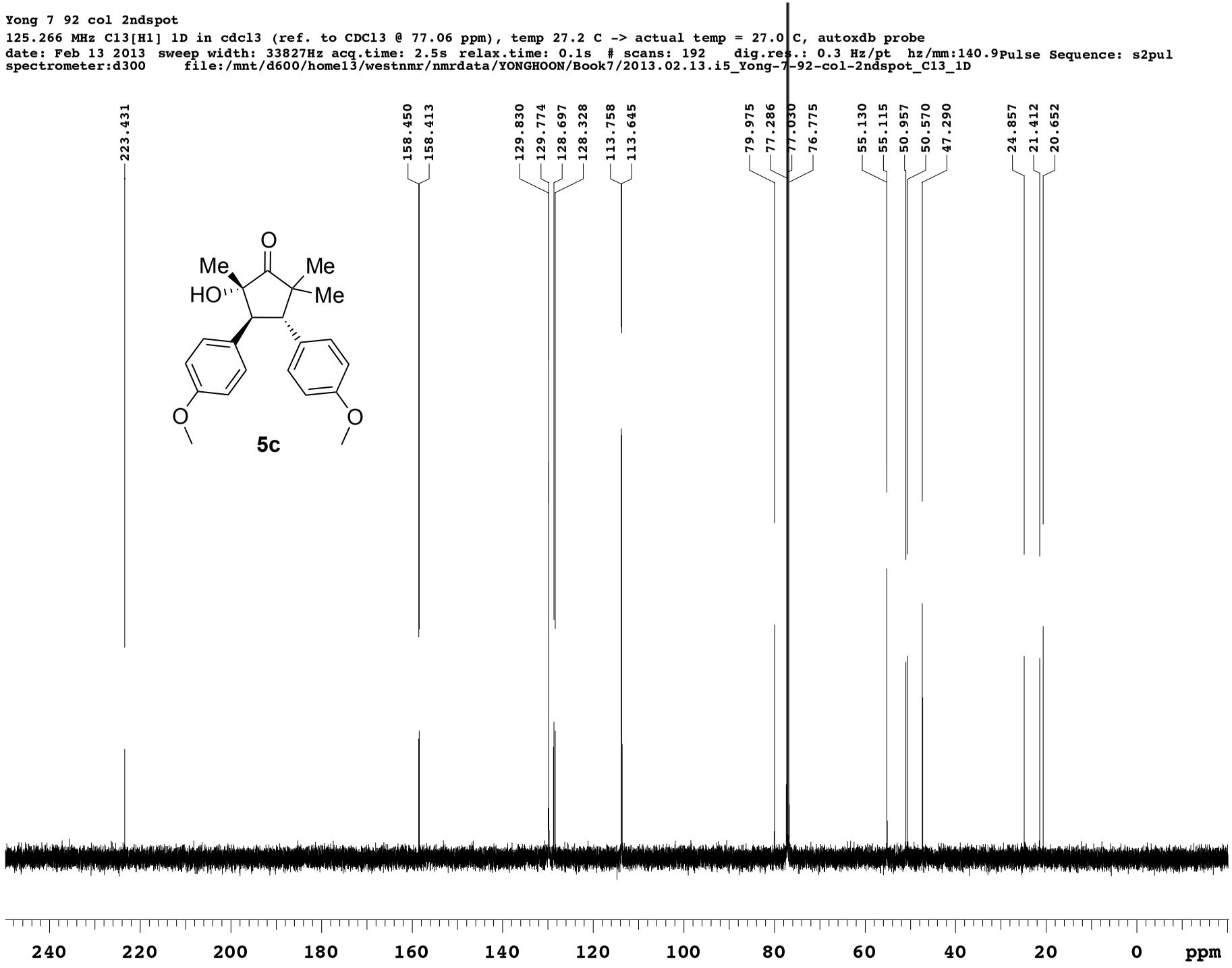
Yong-7-92-col-2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



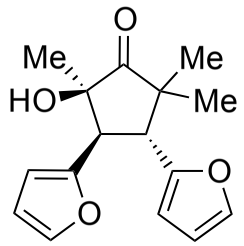
5c

Yong 7 92 col 2ndspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 13 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 192 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.13.i5_Yong-7-92-col-2ndspot_C13_1D



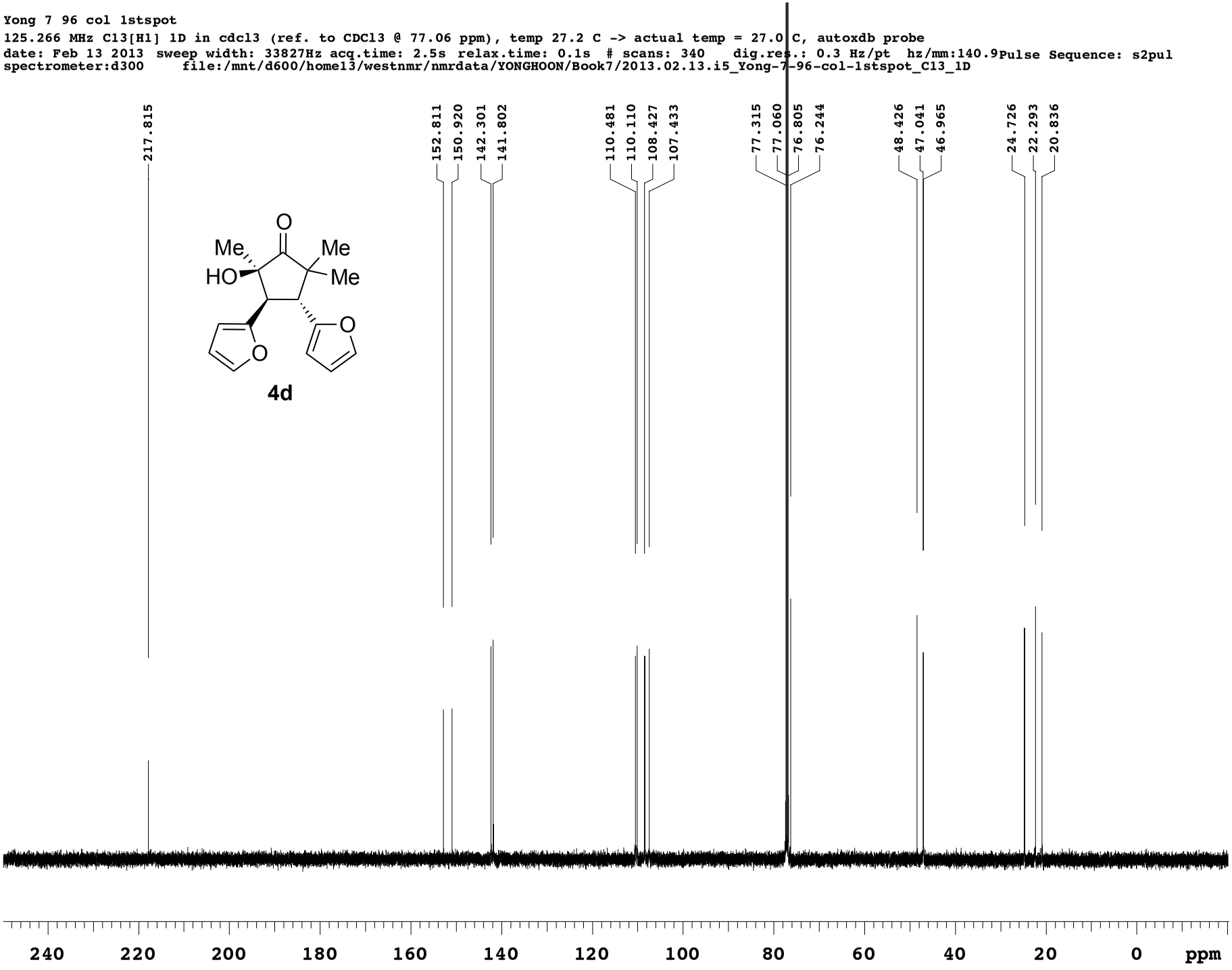
Yong-7-96-col-1stspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



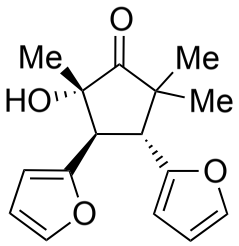
4d

Yong 7 96 col 1stspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 13 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 340 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.13.i5_Yong-7-96-col-1stspot_C13_1D



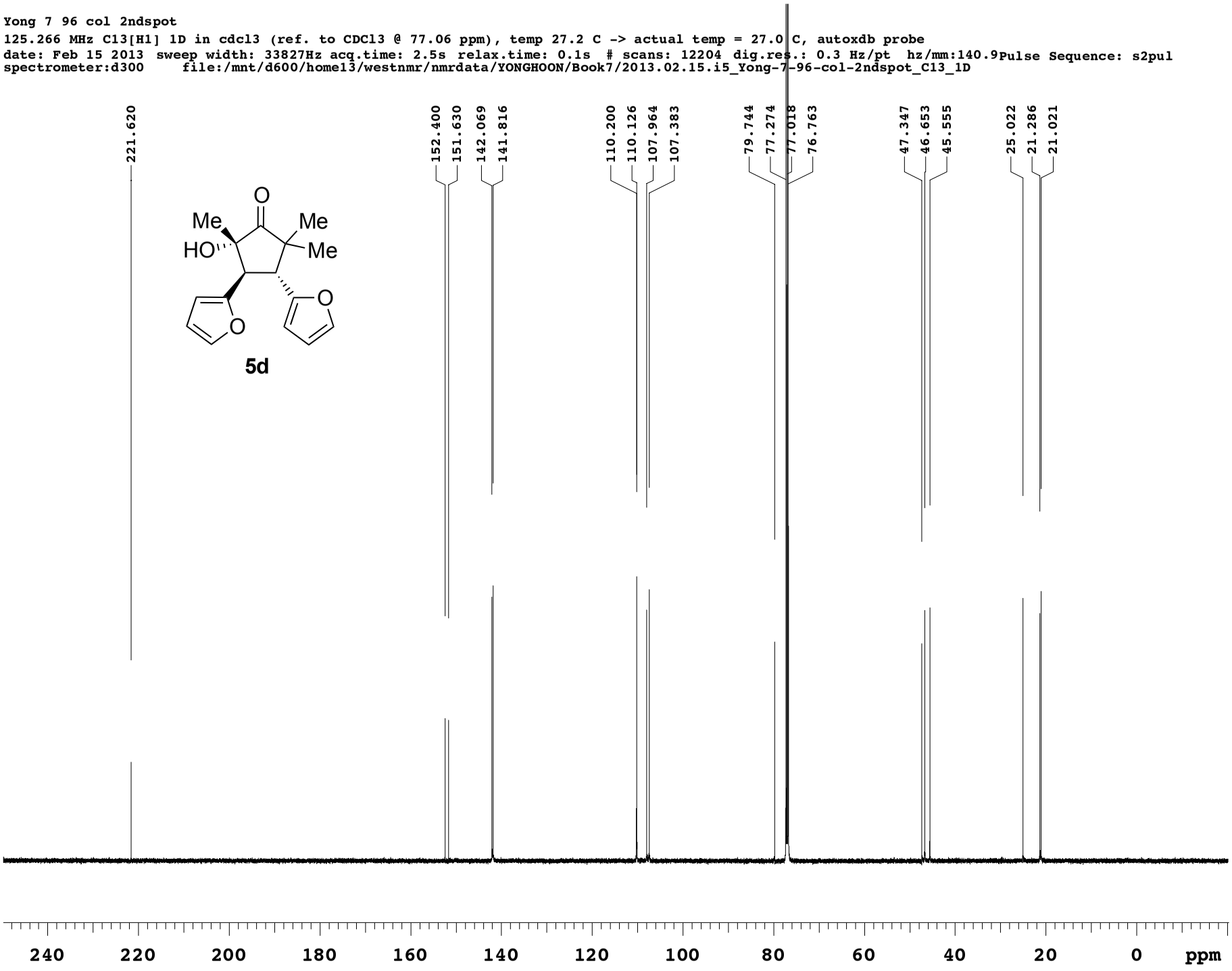
Yong-7-96-col-2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



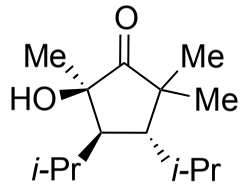
5d

Yong 7 96 col 2ndspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 15 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 12204 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.15.i5_Yong-7-96-col-2ndspot_C13_1D

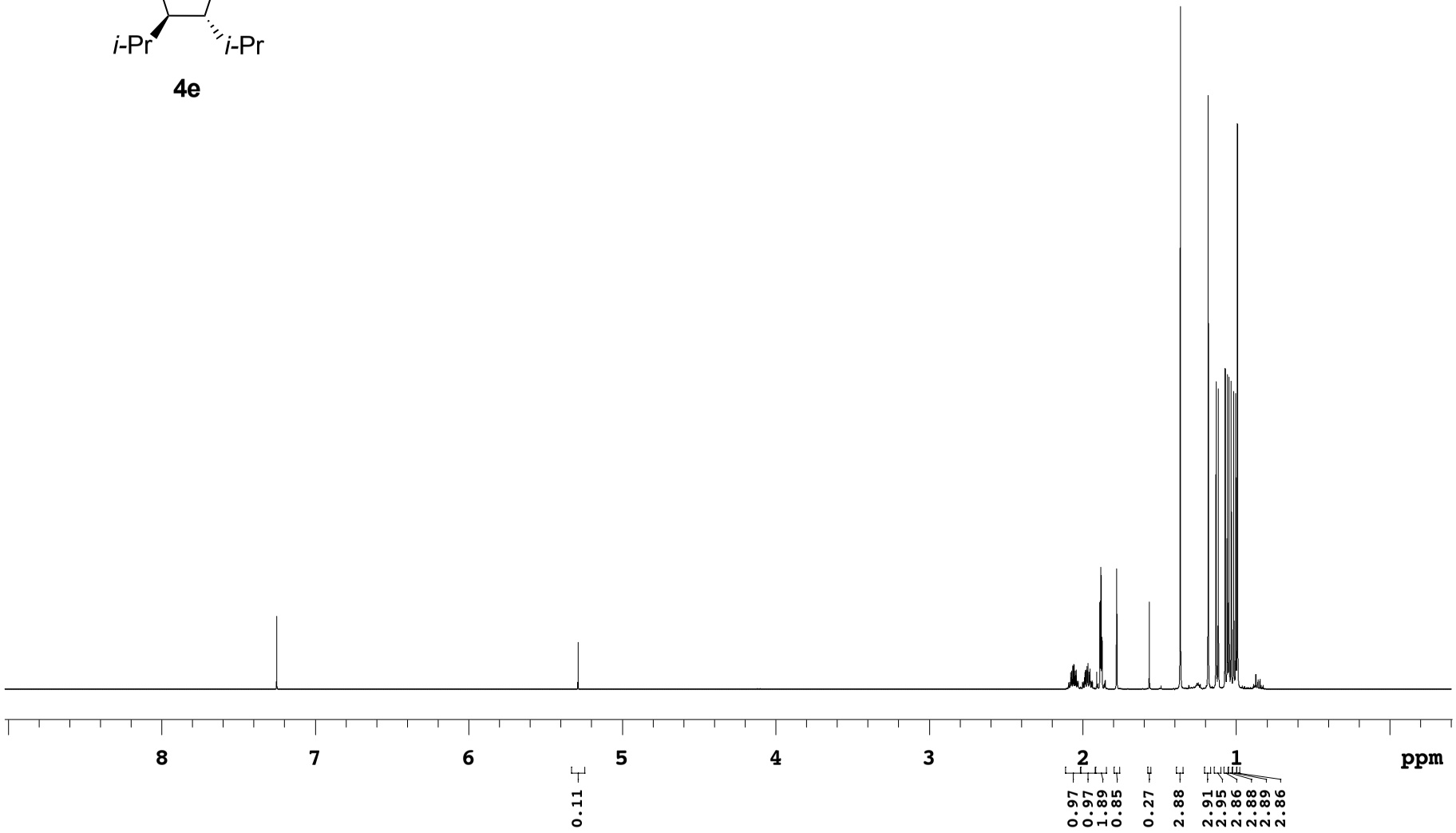


Yong-7-120-col-2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

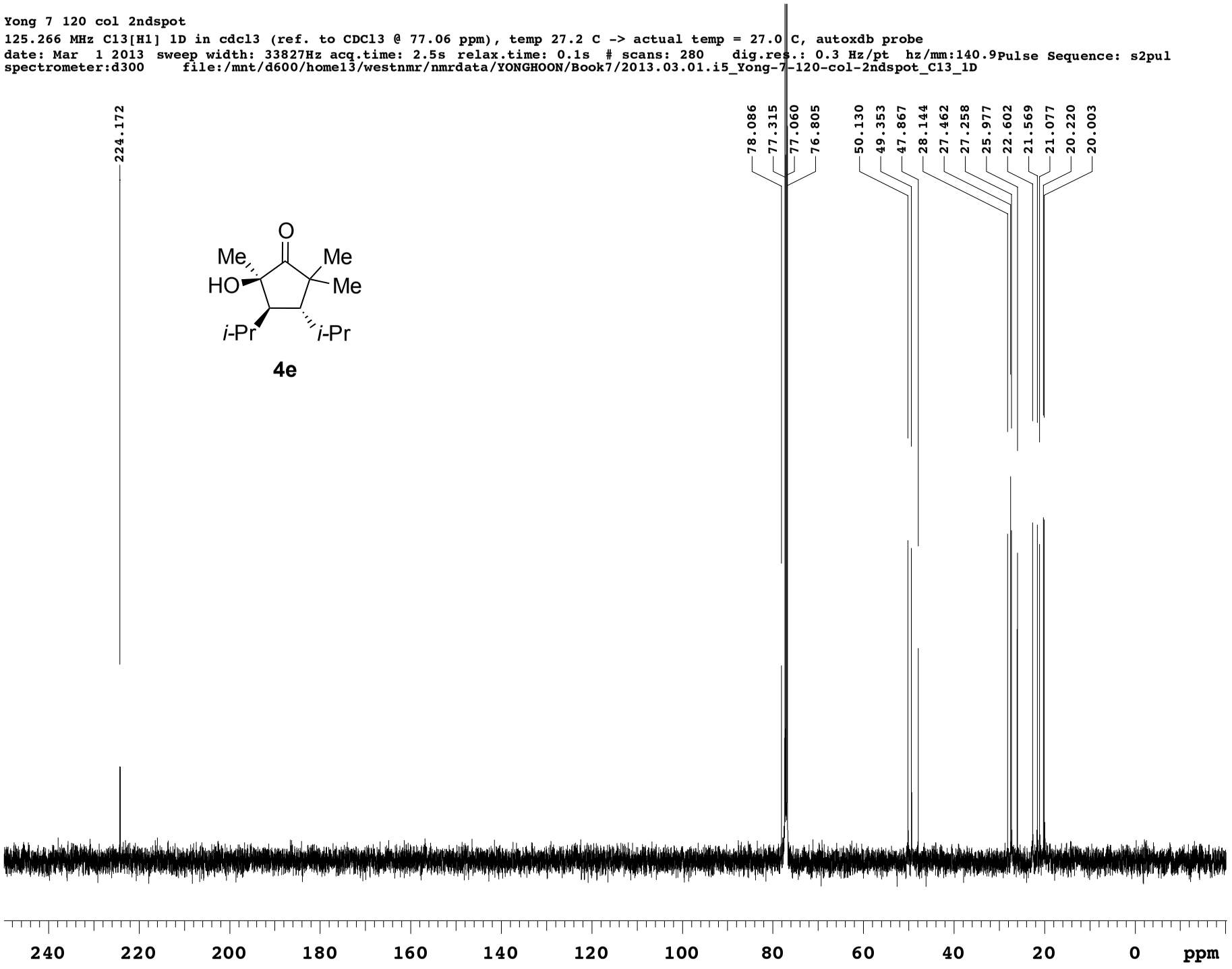
Pulse Sequence: s2pul



4e



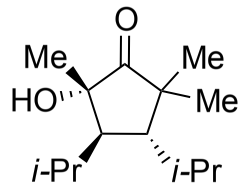
Yong 7 120 col 2ndspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Mar 1 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 280 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.03.01.i5_Yong-7-120-col-2ndspot_C13_1D



4e

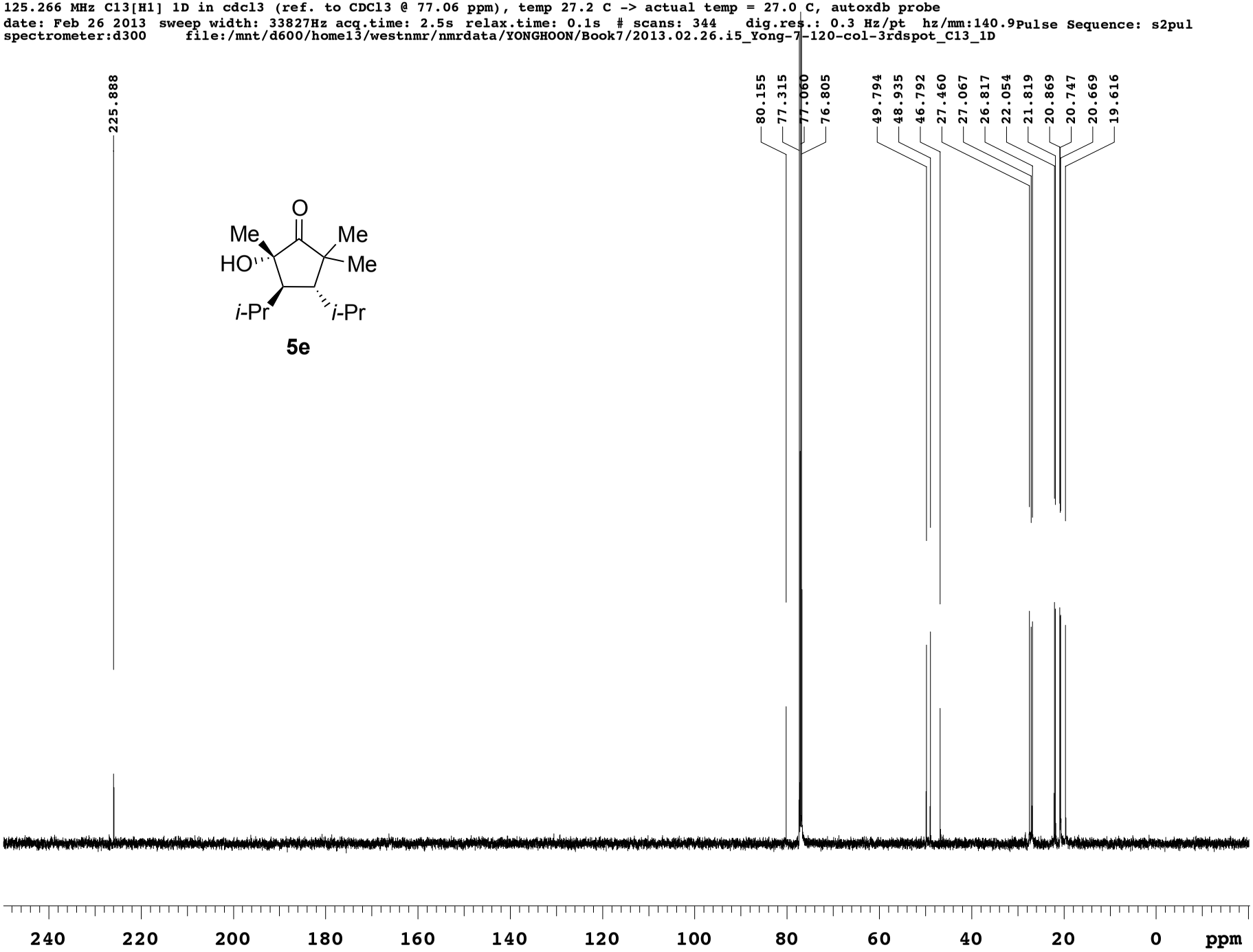
Yong-7-120-col-3rdspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



5e

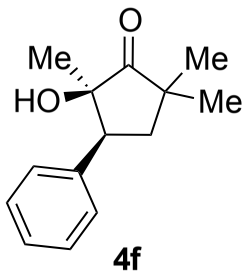
Yong 7 120 col 3rdspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 26 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 344 dig.res.: 0.3 Hz/pt hz/mm:140.9Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.26.i5_Yong-7-120-col-3rdspot_C13_1D



5e

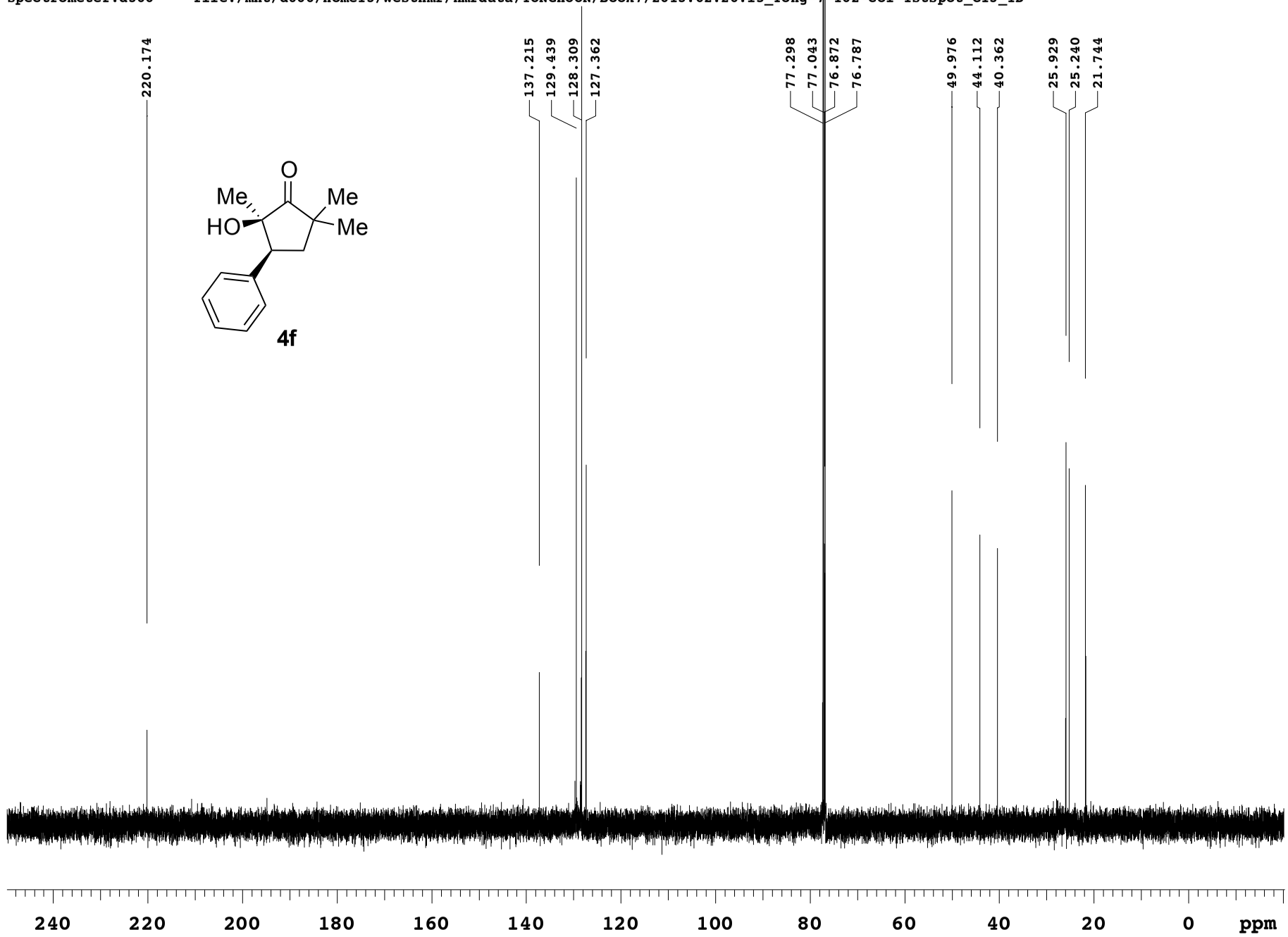
Yong-7-102-col-1stspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul

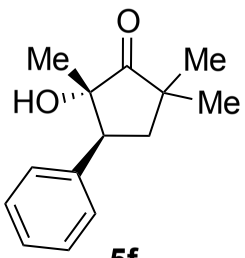


4f

Yong 7 102 col 1stspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 20 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 48 dig.res.: 0.3 Hz/pt hz/mm:140.9Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.20.i5_Yong-7-102-col-1stspot_C13_1D

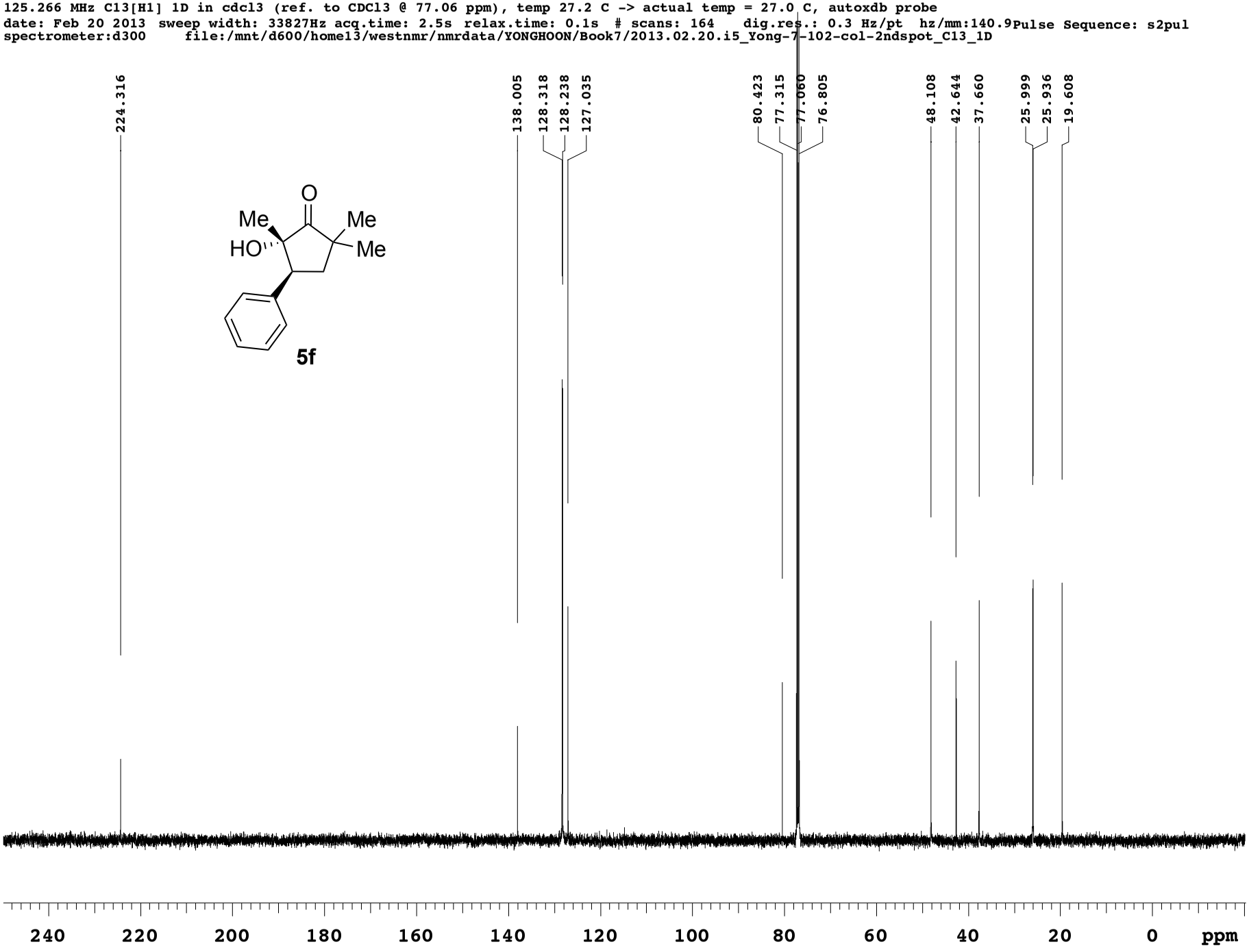


Yong 7 102 col 2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 20 2013 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.5 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.20.i5_Yong-7-102-col-2ndspot_H1_1D



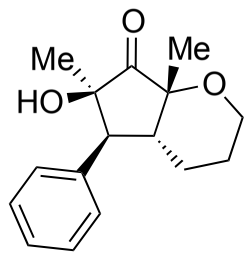
5f

125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 20 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 164 dig.res.: 0.3 Hz/pt hz/mm:140.9Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.20.i5_Yong-7-102-col-2ndspot_C13_1D



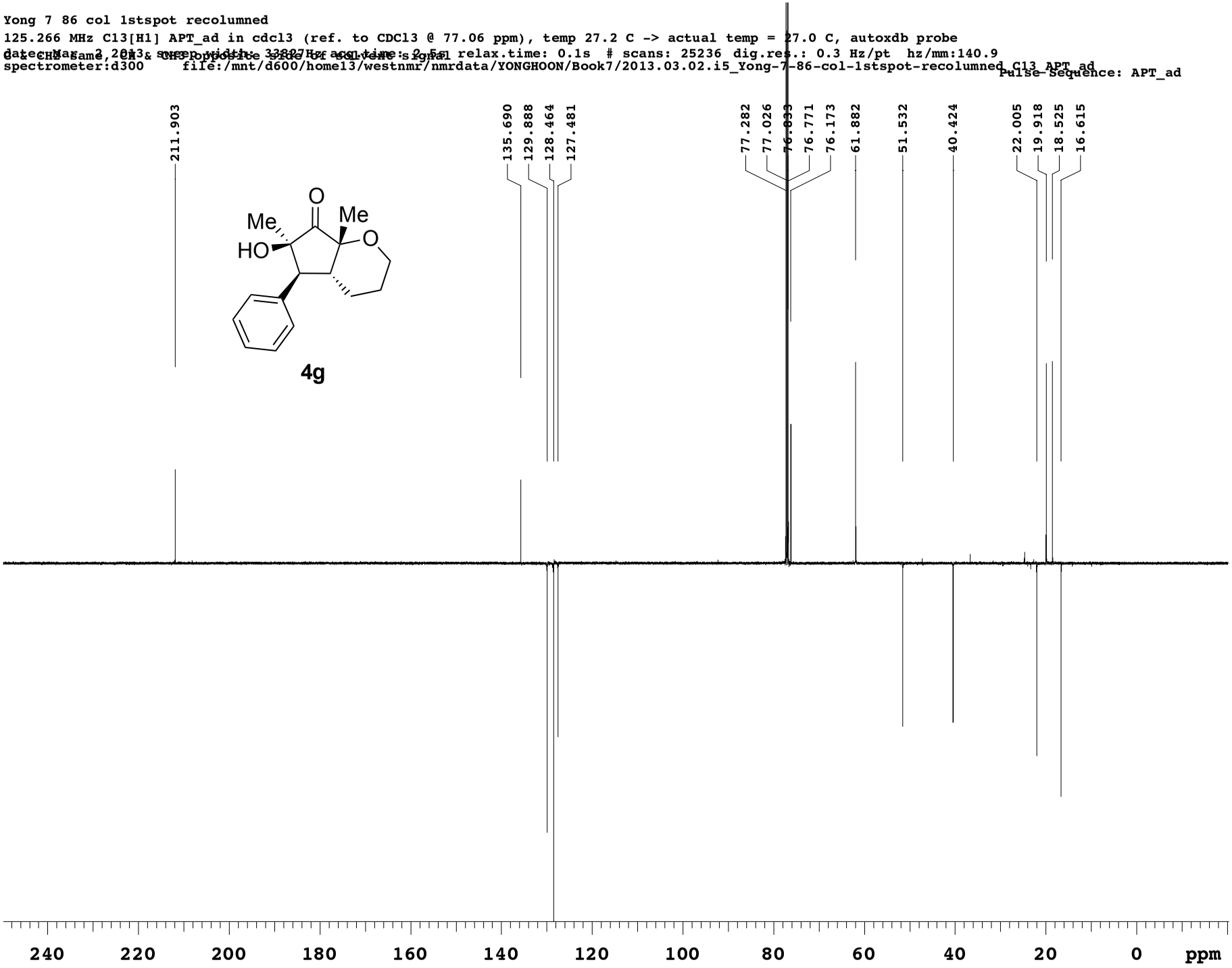
Yong-7-86-col-1stspot-recolumned
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



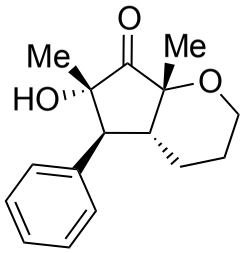
4g

Yong 7 86 col 1stspot recolumned
125.266 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date_Mar 2 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 25236 dig.res.: 0.3 Hz/pt hz/mm:140.9
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.03.02.i5_Yong-7-86-col-1stspot-recolumned_C13_APT_ad
Pulse Sequence: APT_ad



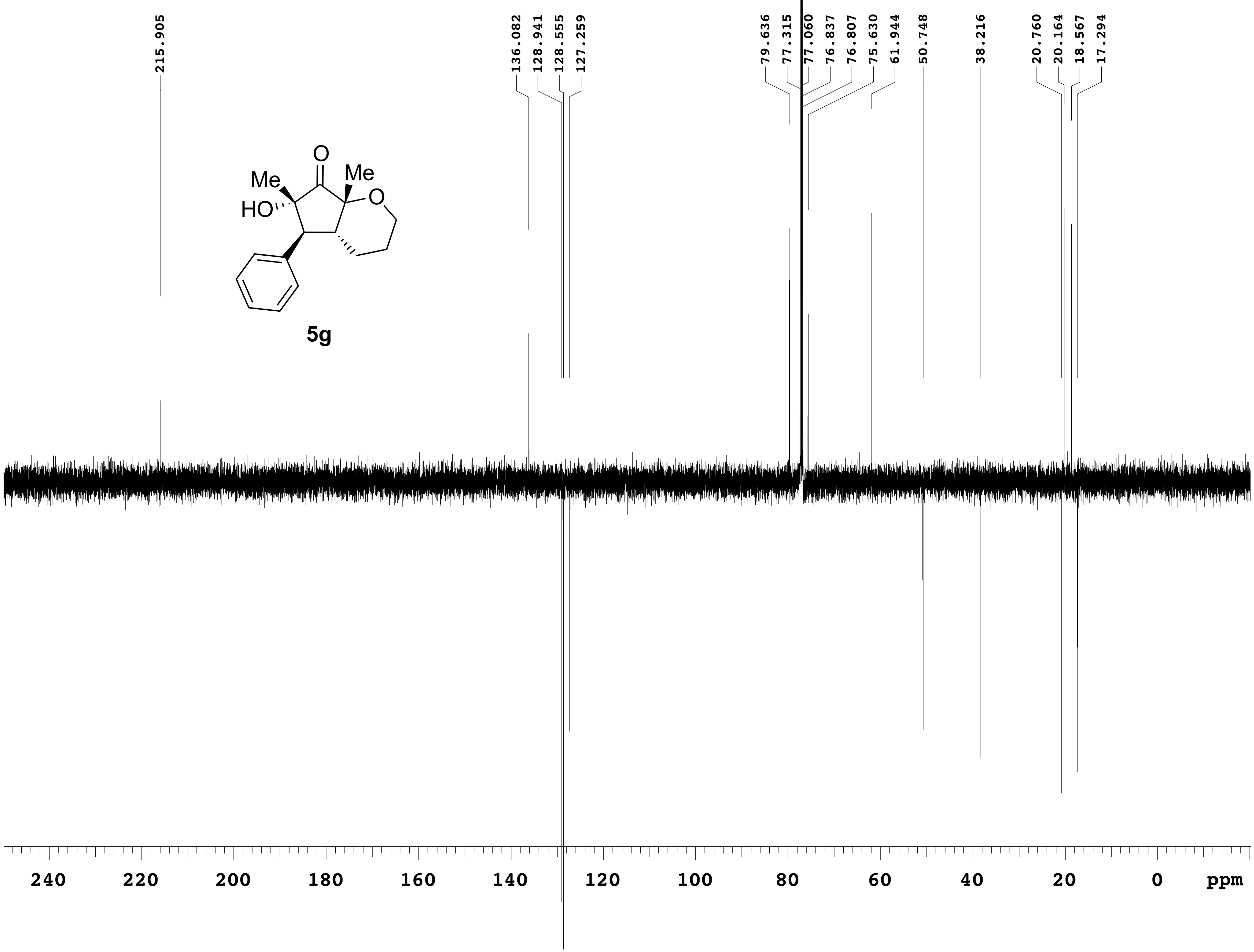
Yong-7-86-col-2ndspot-EAremoved
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



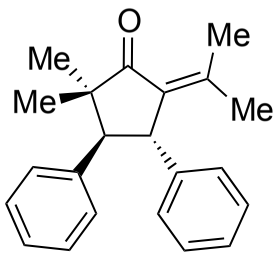
5g

Yong 7 86 col 2ndspot
125.266 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Feb 9 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 240 dig.res.: 0.3 Hz/pt hz/mm:140.9
C & CH2 same, CH & CH3 opposite side of solvent signal
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.02.09.i5_Yong-7-86-col-2ndspot_C13_APT_ad
Pulse Sequence: APT_ad

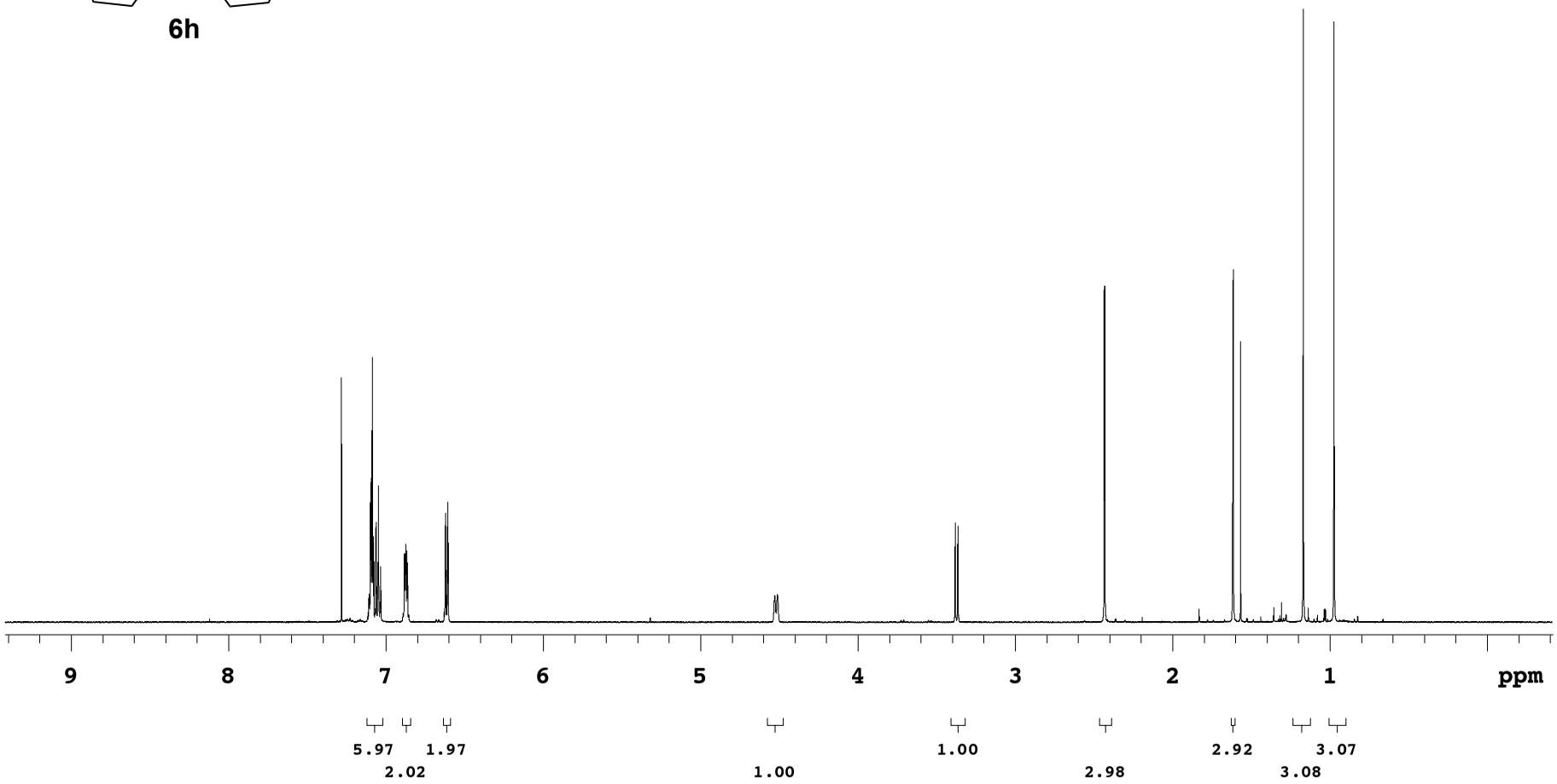


5g

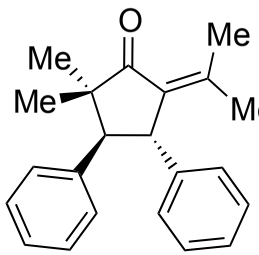
Yong 7 190 col pure
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, autoxdb probe
date: May 8 2013 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.4 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.05.08.i5_Yong-7-190-col-pure_H1_1D



6h



Yong 7 190 col pure
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 26.4 C -> actual temp = 27.0 C, autoxdb probe
date: May 8 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 616 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.05.08.i5_Yong-7-190-col-pure_C13_1D



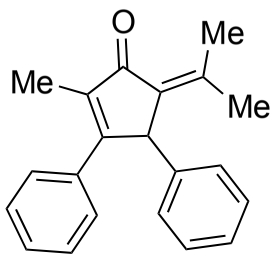
6h

211.291
152.946
141.301
138.196
132.449
130.819
129.581
127.557
127.242
126.305
126.006
77.271
77.016
76.763
57.561
50.498
49.624
26.528
25.461
22.224
21.618

240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

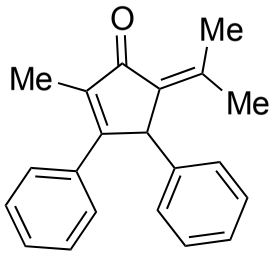
Yong-7-132-col-2ndspot
498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



7h

Yong 7 132 col 2ndspot
125.266 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 26.4 C -> actual temp = 27.0 C, autoxdb probe
date: Mar 18 2013 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 304 dig.res.: 0.3 Hz/pt hz/mm:140.9 Pulse Sequence: s2pul
spectrometer:d300 file:/mnt/d600/home13/westnmr/nmrdata/YONGHOON/Book7/2013.03.18.i5_Yong-7-132-col-2ndspot_C13_1D



7h

197.509
162.378
147.252
140.865
139.393
135.256
134.454
128.527
128.413
128.271
128.219
128.147
126.445
77.315
77.060
76.805
52.509
24.021
20.652
9.897

240 220 200 180 160 140 120 100 80 60 40 20 0 ppm